भारतीय मानक
Indian Standard

IS 4471 : 2022

वस्त्र रंजक सामग्री — नैफ्थॉल (एज़ोईक युग्मक घटक) की तीव्रता के निर्धारण की विधि (भारात्मक एवं आयतनात्मक विधि)

(दूसरा पुनरीक्षण)

Textile Dyestuffs — Methods for Determination of Strength of Naphthols (Azoic Coupling Components) (Gravimetric and Volumetric Methods)

(Second Revision)

ICS 59.040; 71.040.50

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October 2022 **Price Group 4**

Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Textile Speciality Chemicals and Dyestuffs Sectional Committee had been approved by the Textiles Division Council.

This standard was first published in 1967. It was observed by the industry that the gravimetric method given in the standard did not give consistent results and some naphthols were not getting dissolved completely. First revision of this standard was undertaken in 1981 to include volumetric method for strength determination of napthols in addition to gravimetric method already given in standard.

The present revision has been made in light of experience gained since its last revision to incorporate the following major changes:

a) Title of the standard has been modified;
b) Grade and purity of chemicals used have been specified;
c) Sampling clause has been modified; and
d) References to Indian Standard has been updated.

The composition of the Committee responsible for the formulation of this standard is given in Annex B.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Indian Standard

TEXTILE DYESTUFFS — METHODS FOR DETERMINATION OF STRENGTH OF NAPHTHOLS (AZOIC COUPLING COMPONENTS) (GRAVIMETRIC AND VOLUMETRIC METHODS)

(Second Revision)

1 SCOPE

1.1 This standard prescribes two methods, namely, gravimetric and volumetric, for determination of strength of naphthols. Both the methods are applicable to the naphthols given in Annex A.

1.2 Volumetric method prescribed in the standard is not applicable to the mixtures of naphthols.

1.2.1 In case of dispute, the gravimetric method shall be used for determination of strength of naphthols.

2 REFERENCES

The following standard contains provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below.

IS No.	*Title*
517 : 2020	Specification for methanol (Methyl alcohol) (*third revision*)
1070 : 1992	Reagent grade water – Specification (*third revision*)

3 SAMPLING

3.1 Lot — All the containers of the same naphthol and of the same strength delivered to a buyer against a dispatch note shall constitute a lot.

3.2 Unless otherwise agreed to between the buyer and the seller the number of containers to be selected at random from a lot shall be in accordance with Table 1.

3.3 From each container draw small quantities of the naphthol by a suitable sampling instrument from at least three different parts and mix them thoroughly to get a composite sample weighing about 50 g. This shall constitute the test sample.

Table 1 Sample Size

(*Clause* 3.2)

Lot Size (1)	**Sample Size** (2)
2 to 15	2
16 to 25	3
26 to 50	4
51 to 100	5
101 to 150	6
151 to 300	7
301 and above	8

4 QUALITY OF REAGENTS

4.1 Unless otherwise specified analytical reagent grade chemicals with 99.0 percent purity shall be employed in tests and distilled water (*see* IS 1070) shall be used where the use of water as reagent is intended.

5 GRAVIMETRIC METHOD

5.1 Apparatus

5.1.1 Beaker — of 1 000 ml capacity.

5.1.2 *Sintered Glass Funnel* — of appropriate size:
with pore size 15 to 40 microns (G-3 size), and
with pore size 5 to 15 microns (G-4 size).

5.2 Reagents

5.2.1 *Methanol* —. Pure (*see* IS 517).

5.2.2 *Sodium Hydroxide Solution* — 30 percent (*m/m*) and 1 percent (*m/m*).

5.2.3 *Hydrochloric Acid* — 1:1 (*v/v*).

5.3 Procedure

5.3.1 Weigh accurately about 1 g of naphthol sample and transfer to 1000 ml dry beaker. Paste it with 10 ml methanol and add 1.5 ml of 30 percent sodium hydroxide and stir to dissolve. Add 400 ml warm (about 50°C) 1 percent sodium hydroxide solution and stir till the solution becomes clear. Filter through sintered glass funnel G-3, wash the residual material first with 3 to 5 ml methanol and finally with two 25 ml portions of warm 1 percent sodium hydroxide solution. Transfer the filtrate and washing quantitatively into a 1 000 ml beaker. Add while stirring with a glass rod, hydrochloric acid, drop by drop until the solution is acidic to Congo red paper. Digest the precipitated mass on water-bath for about 2 to 3 h. Allow the precipitate of naphthol to settle down. Filter through a sintered glass funnel G-4. Wash the precipitate with hot water till the filtrate is free from chlorides.

5.3.2 Dry the residue at 100 to 110°C to constant weight and weigh.

5.3.3 Calculate the percentage of naphthol by the following formula:

$$p = \frac{b}{a} \times 100$$

where

p = percentage, by mass of the naphthol under test

b = mass, in g, of the residue of the naphthol; and

a = mass, in g, of the naphthol taken for test.

5.4 Repeat the test twice as given in **5.3.1** and **5.3.2** and calculate the percentage of naphthol by the formula given in **5.3.3**.

5.5 Calculate the average of the values obtained as in **5.3.3** and **5.4**.

6 VOLUMETRIC METHOD

6.1 Apparatus

6.1.1 *Amber-Coloured Jacketed Burette*

6.1.2 *Amber-Coloured Graduated FIask* — of 500 ml capacity.

6.1.3 *Mechanical Stirrer*

6.1.4 *Beakers* — of 1 000 and 500 ml capacities.

6.1.5 *Water-Bath*

6.2 Reagents

6.2.1 *Para Amino Acetanilide Diazonium Chloride Solution (Diazo Solution)* — 0.05 N, prepared as follows:

Weigh accurately 3.77 g of para amino acetanilide and transfer it to a 500 ml beaker. Add 55 ml of 1 N hydrochloric acid and 100 ml of water. Cool externally with ice to 5 to 10°C while stirring mechanically. Add 25.5 ml of 1 N sodium nitrite solution in about 5 min time. Continue stirring for 10 min and test for excess of nitrite with starch iodide paper (*see* Note). Transfer the solution to a 500 ml amber coloured graduated flask and make up the volume with ice cold water. Keep the diazo solution in ice-bath.

NOTE — The starch iodide paper shall give a positive faint test of excess of nitrous acid.

6.2.2 *Diazotized Aniline Solution* — 0.05 N, prepared as follows:

Weigh accurately 2.325 g of redistilled aniline and diazotize it in the same manner as for para amino acetanilide (*see* **6.2.1**).

NOTE — This solution shall be used for estimating the strength of C. I. Azoic Coupling Component 18.

6.2.3 *Dianisidine Diazonium Chloride* — 0.02 N, prepared as follows:

Diazotize 5 g to the dianisidine base with 10 ml of hydrochloric acid and 3 g of solid sodium nitrite. Neutralize the solution with a fresh sodium acetate solution and dilute it to 1000 ml.

6.2.4 *H-Acid (1-Naphthol-3.6, Disulphonic Acid, 8 Amino)* — 0.1 percent, prepared as follows:

Dissolve 0.1 g of H-acid in 100 ml of water containing 5 ml of 2 N sodium carbonate.

6.2.5 *Hydrochloric Acid* — 1 N and 0.1 N.

6.2.6 *Sodium Hydroxide Solution* — 1 N.

6.2.7 *Dilute Sodium Hydroxide Solution* — 2.5 ml of 40 percent sodium hydroxide in 100 ml of water.

6.2.8 *Dilute Acetic Acid* — 25 percent (*m/v*).

6.2.9 *Pyridine*

6.3 Procedure

6.3.1 Take about 1 g of naphthol from the composite sample. Weigh it accurately and dissolve it in 40 ml of dilute sodium hydroxide solution (*see* **6.2.7**). Heat the solution, if necessary, to dissolve the naphthol completely, Cool and add dilute acetic acid till the *p*H of the solution is neutral (*p*H 6 to 7). Dissolve the precipitated naphthol in 80 ml of pyridine with constant stirring. Add more pyridine, if necessary. Cool it to 25°C and keep under mechanical agitation.

6.3.2 Titrate the solution obtained as in **6.3.1** against diazotized para amino acetanilide solution (diazo solution) (*see* **6.2.1**) from an amber coloured burette equipped with a water jacket through which water at about 5°C is circulated. Spot a drop of the reaction mixture on a filter paper. Spot a drop each of dianisidine diazonium chloride solution and H-acid solution near the spot of the reaction mixture. Take the end-point as the mean of the two readings, one which gives a negative test for naphthol with dianisidine diazonium chloride and the other which gives a positive test for para amino acetanilide diazonium chloride solution with H-acid. Note the volume of the para amino acetanilide diazonium chloride solution required.

NOTES

1 The para amino acetanilide diazonium chloride solution should be added initially in portions of 1 to 2 ml, testing the reaction mixture after each addition for excess of diazonium chloride or naphthol.

2 If the bleed of reaction mixture when spotted on filter paper is not clear, a small amount of solid common salt should be kept on filter paper and the drops of reaction mixture should be dropped on it.

3 If naphthol is in excess, a colouration is seen at the junction of the spots of dianisidine diazonium chloride and the reaction mjxture. If diazo solution is in excess, a pink colour is seen at the junction of the spot of H- acid and the reaction mixture. The addition of para amino acetanilide diazonium chloride solution should be in instalments of 0.2 to 0.3 ml near the end point.

4 In case of Azoic Coupling Component 18, diazotized aniline solution (*see* **6.3.2**) should be used.

5 C I Azoic Coupling Component 5 is dissolved in pyridine at 40°C. No alkali is added. The solution is cooled to 5 to 10°C and titrated against diazotized para amino acetanilide.

6.3.3 Calculate the strength of naphthol by the following formula:

$$P = \frac{0.005 \times M \times A \times 100}{B}$$

6.3.4 In case of Azoic Coupling component 5, the coupling takes place at two positions. Therefore, the amount of diazo solution consumed is double and hence the strength should be calculated as:

$$P = \frac{0.005 \times M \times A}{2} \times \frac{100}{B}$$

where

P = percentage, by mass, of naphthol;

M = molecular mass of naphthol under test (*see* Annex A); and

A = volume, in millilitres, of para acetanilide diazonium chloride solution (or diazotized aniline solution) required for titration; and

B = mass, in g, of the naphthol taken for the test (*see* **6.3.1**).

6.4 Repeat the procedure twice prescribed in **6.3.1** and **6.3.2** and calculate the strength of naphthol in each case.

6.5 Calculate the average of the values obtained as in **6.3.3** and **6.4**.

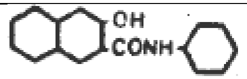
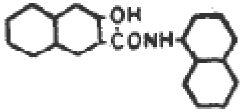
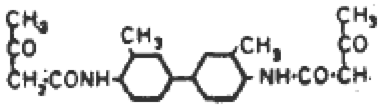
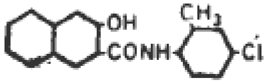
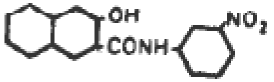
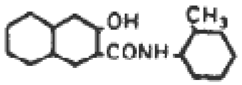
7 REPORT

7.1 Report the value obtained as in **5.5** or **6.5** as the strength of naphthol under test.

7.2 Report also the method used, whether gravimetric or volumetric.

ANNEX A
GENERAL INFORMATION ABOUT ANALYSIS OF NAPHTHOLS

(*Foreword and Clauses* 1.1 *and* 6.3.3)

Sl No. (1)	**C I Designation* of Naphthol** (2)	**Common Commercial Name of Naphthol** (3)	**Colour Index No.** (4)	**Molecular Mass** (5)	**Structural Formula and Chemical Name** (6)
i)	C I Azoic Coupling Component 2	Naphthol AS	C. I. 37505	263.0	
ii)	C I Azoic Coupling Component 4	Naphthol AS-BO	C. I. 37560	313.0	
iii)	C I Azoic Coupling Component 5	Naphthol AS-G	C. I. 37610	380.0	
iv)	C I Azoic Coupling Component 8	Naphthol AS-TR	C. I. 37525	311.5	
v)	C I Azoic Coupling Component 17	Naphthol AS-BS	C. I. 37515	308.0	
vi)	C I Azoic Coupling Component 18	Naphthol AS-D	C. I. 37520	277.0	

*Colour Index (1956) Ed 2. Society of Dyers and Colourists, UK; and American Association of Textile Chemists and Colorists, USA

ANNEX B
(*Foreword*)

COMMITTEE COMPOSITION

Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

Organization	*Representative(s)*
Department for Jute and Fibre Technology Institute of Jute Technology, University of Calcutta, Kolkata	Prof A K Samanta (***Chairman***)
Ahmedabad Textile Industry's Research Association,	Shrimati Deepali Plawat Shrimati Fahimunnisa Khatib (*Alternate*)
Ama Herbals Laboratories Pvt Ltd, Lucknow	Shri Y A Shah
Archroma India Pvt Limited, Mumbai	Shri Rajesh Ramamurthy Shri Ashim Ghosh (*Alternate*)
Atul Limited (Colors Business), Valsad	Shri V R Sai Ganesh Shri Arindam Chakraborty (*Alternate*)
Bio Dyes India Pvt Ltd, Goa	Dr Bosco Henriques
Central Coir Research Institute, Alappuzha	Shrimati Anita Jacob Shrimati Sumi Sabestian (*Alternate*)
ICAR – Central Institute for Research on Cotton Technology, Mumbai	Dr Sujata Saxena Dr A S M Raja (*Alternate*)
Department for Jute and Fibre Technology Institute of Jute Technology, University of Calcutta, Kolkata	Dr D Das
Global Organic Textile Standard, (GOTS), Thane	Shri Rahul Bhajekar Ms Prachi Gupta (*Alternate*)
Indian Jute Industries Research Association, Kolkata	Dr S K Chakrabarti Shri Sandip Basu (*Alternate*)
Northern India Textile Research Association, Ghaziabad	Dr M S Parmar
Office of the Textile Commissioner, Mumbai	Shri Gaurav Gupta Shri Sanjay Charak (*Alternate*)
SGS India Pvt Ltd, Mumbai	Shri Karthikeyan K Shri Gaurav Saraswat (*Alternate*)
Shree Pushkar Chemicals & Fertilizers Ltd, Mumbai	Dr N N Mahapatra
Textiles Committee, Mumbai	Shri Kartikeya Dhanda Shrimati Shilpi Chauhan (*Alternate*)

Organization	*Representative(s)*
The Arvind Mills Limited, Ahmedabad	Shri Rajarshi Ghosh Shri Umasankar Mahapatra (*Alternate*)
The Bombay Textile Research Association, Mumbai	Dr Padma S Vankar Shri M P Sathianarayanan (*Alternate*)
The South India Textile Research Association, Coimbatore	Dr Prakash Vasudevan Shri S Sivakumar (*Alternate*)
The Synthetic and Art Silk Mills Research Association, Mumbai	Shrimati (Dr) Manisha Mathur Shrimati Ashwini Sudam (*Alternate*)
U P Textile Technology Institute, Kanpur	Dr Arun Patra
Wool Research Association, Thane	Shrimati Smita Bait Shrimati (Dr) Mrinal Choudhari (*Alternate*)
BIS Directorate General	Shri J K Gupta, Scientist E and Head (Textiles) [Representing Director General (*Ex-officio*)]

Member Secretary
Shri Himanshu Shukla
Scientist B (Textiles), BIS

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Review of Indian Standards

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